

EVALUATION OF SPECIMEN DISPLACEMENT ERRORS IN HIGH TEMPERATURE POWDER DIFFRACTION FURNACES

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Requirements for accurate measurement of unit cell parameters in high temperature powder diffraction furnaces are described in detail and results are shown for several materials. The effects of the primary sources of systematic errors, which include sample displacement and temperature measurement, are demonstrated for two materials using the internal standard calibration and Rietveld methods.

The paper will focus on the use of resistively heated metal strip furnaces, which are prone to sample displacement that changes with temperature. Results are shown for a Buehler HDK2 diffraction furnace that employs two metal strip heaters: one as the sample support and the second as an “environmental” heater that surrounds the specimen.

The traditional internal calibration method is applied using several levels of data calibration for measurements of Al_2O_3 , while the Rietveld whole-pattern fitting method is applied for analysis of $\text{BaZr}_4\text{P}_6\text{O}_{24}$. The data analysis provides a description of the specimen displacement as a function of temperature and shows the inaccuracies encountered when specimen displacement is not corrected. The data demonstrate that special care must be taken to eliminate systematic instrumental errors. Diffraction pattern calibrations are critical for accuracy, and can be performed using the traditional internal standard method or using Rietveld refinement.

The use of NIST temperature calibration standards will be described briefly, and the applicability of the HDK2 diffraction furnace for large monolithic specimens will be demonstrated.

Finally, the performance of a custom diffraction furnace will be compared to data from the strip heater furnace. The custom diffraction furnace was designed to minimize systematic errors in temperature and XRD line position. The data show that reliable unit cell parameters can be obtained from RT to 1450 °C without calibrating the diffraction patterns.